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EMERGING MATERIALS TECHNOLOGY IN JAPAN

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TECHNOLOGY MANAGEMENT BRANCH

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US ARMY
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ABSTRACT

Remarkable progress has been made in Japan on new materials in processing, characterization, standardization of test methods, design and analysis, and systems applications. In the past four years basic research activity in universities, government laboratories, and industry has accelerated. The production base has continued to grow. A Ministry of International Trade and Industry survey in 1987 estimated that approximately 29,000 engineers and scientists were engaged in applications of new materials in product development. Japan has made notable progress in many important materials technologies. Japanese technologists have a strong commitment to develop new materials and have been following through on this commitment since the late 1970s. This is especially so in advanced composites, and particularly in fine ceramics. Japan has a large investment in pilot plants where the market is as yet unproven and currently unprofitable. Their leadership is unquestioned in small-lot production using advanced materials, and their pursuit of thin markets is notorious among competitors. This often gives them early market dominance. For instance, many U.S. firms make ceramics with Japanese powders and composites with Japanese fibers and whiskers. Japanese industry is also investing heavily in new materials. They have the financial support and technical cooperation of the government in planning for the future. Accordingly, there is a coherent and long-term national strategy with well disciplined and strong corporate commitment. In many areas the U.S. and Japan are on par in materials science and basic technology, but Japan is advancing more rapidly into commercial applications and in recent years has emphasized basic research. Therein lies a powerful challenge to the U.S. materials community. Japan has come a long way in materials technology in the past four years and poses a very real threat to U.S. technological leadership. Emerging materials technology in Japan now requires constant attention from the U.S. community.

The following topics are briefly covered in the Executive Summary but are discussed in detail in the body of this report.

- Powder Technology
- Net Shaping and Innovative Processing
- Higher Strengths, Improved Fracture Toughness and Tailored Properties: Sialons, SiC, Si_3N_4, Zirconia, Mullite
- Carbon/Carbon Materials
- Machining and Ceramics
- Specifications and Standards
- Development of the Infrastructure: Ceramics Technology Base

EXECUTIVE SUMMARY

Progress In Overcoming Materials Technology Barriers

Since the early 1970s many billions of dollars have been spent by the United States for developing structural ceramics and advanced composite materials. Yet the major research and development (R&D) needs and materials technology barriers remain the same. There is continued need for improved and specifically tailored mechanical, thermal, electrical, and other properties, especially at elevated temperatures. There is need for improved reliability and reproducibility, better designs, and improved characterization methods for new materials, all at lower costs.

Structural Ceramics

In response to these technological requirements, particularly in Japan, there has been a move to develop alternate powder processing routes to produce higher purity, smaller particle size powders, and "composite" powders. Powders of sub-micron size open routes to achieving a wider range of mechanical and physical properties, and to important characteristics such as "superplasticity." Such powders also point the way to new routes to microstructural control. In manufacturing, finer powders often result in faster sintering times and lower production costs. Other major thrusts include: processing under extreme conditions (high pressures, dynamic compaction, self-propagating combustion synthesis (SHS), etc.); systematic exploration of sintering aids: new combination of materials and novel processing techniques. Motivations for the Japanese work are to improve properties, lower costs, use stocks of available raw materials, and to break existing patent stalemates and achieve a viable patent situation.

Powder Technology

Starting in the early 1980s, Japanese academic, government, and industrial researchers cooperated to improve powder technology using a wide range of materials precursors and processing techniques. Akio Kato of Kyushu University was active in the early development of SiC and Si_3N_4 powders. He investigated the sinterability of 13 types of commercial Si_3N_4, as well as SiC. He investigated chemical vapor deposition (CVD) and RF-plasma reactor methods for producing ultrafine ceramic powders of high purity. SiC powders received considerable attention. K. Sawano of Nippon Steel used CO_2 Laser syntheses for thermal decomposition of silane-methane gas. M. Endo of Shinshu University used gas-phase pyrolysis in a CVD apparatus based upon a hot walled furnace (700°C to 1400°C), to produce SiC powders of 100 nanometer (nm) average diameter. Numerous investigators have studied RF-plasma syntheses at atmospheric pressure, using mixtures of $SiCl_4/CH_4$ and H_2 to produce cubic SiC with particle sizes in the 10 nm to 20 nm range. These include K. Kijima and M. Konishi of National Institute for Research in Inorganic Materials (NIRIM), Y. Toyama, Japan Fine Ceramics Center (JFCC), A. Mitsui, A. Kato, A. Kuibara, and J. Hojo at Kyushu University, and K. Ishizaki of Nagaoka University of Technology. A hybrid RF-plasma method was studied by K. Akashi to produce beta SiC from an $SiCl_4/CH_4$ mixture carried in H_2. J. Hojo, H. Maeda, and A. Kato of Kyushu University produced amorphous powders of 0.02 micron average diameter beta silicon carbide and alpha silicon nitride in vapor phase reactor using Si $(CH_3)_4$, NH_3, and H_2. Heating at 1500°C crystallized the powders into the beta and alpha forms. Nippon Steel explored CO_2 laser synthesis, and many companies investigated RF or microwave arc processing. SiC powders have been studied by Showa Denko, Nippon Kokan KK Company, Ltd. (NKK), Nissin Flour Milling Company, Hitachi, NGK Spark Plugs, and Kyocera. Ibiden Company produces about 70 tons a month of beta SiC powder of 0.3 micron average size and 98% purity. Mitsui Toatsu sells a high purity beta SiC with an average size of 0.2 microns.

Japanese firms are major suppliers of silicon nitride and have developed numerous processes for high quality powder production. Every important variety of fine ceramic powder is being studied and produced in Japan. Currently, there are over 90 ceramic powder producers. At least seven companies manufacture Al_2O_3, over four make ZrO_2, more than eight supply Si_3N_4, and at least nine companies produce SiC. Still the search for improved powders continues. Professors Miyamoto, Koizumi, and colleagues have been investigating selfpropagating high temperature combustion synthesis (SHS) to synthesize a wide variety of nitride and carbides and their composites. Kyoritsu Company has commercialized SHS powder processes to make powders in quantities of several hundred pounds per month.

Masayuki Horie and coworkers at Tokyo University of Agriculture and Technology, investigated the kinetics of silicon nitridation by silica reduction in the fluidized bed process. Noriasu Hotta and colleagues at Niigata University and Showa Aluminum studied nitridation of Si particles using an air floatation and classification technique to reduce particle sizes. The current price structure of fine ceramics is expected to improve. At present, fine ceramic powders vary widely in price from 1,000 yen/kg* for ferrites or zinc oxide to 100,000 yen/kg for cubic boron nitride (CBN). The majority of powders cost in the range of 10,000 yen/kg. Recently, gas phase synthesis and sol-gel processing have dominated powder processing research. Plasma synthesis has potential for improved chemical homogeneity, high purity, unique microstructural properties, and control over stoichiometry. It is obvious that the end of the line in terms of purity, cost, and varieties of powder precursors has not been reached.

Net Shaping and Innovative Processing

A long standing goal has been to achieve near-net-shaping capability. One innovative approach is use of supercritical fluid extraction (SFE) during the dewaxing process. Sumitomo Heavy Industries, Ltd., Koransha Company, and NKK have all explored SFE techniques. Kiransha uses hard tooling and slip casting, whereas NKK developed a thin film or rubber membrane-lined mold, and cold isostatic pressuring along with SFE.

NKK has produced impressive high performance large parts (about 25 cm diameter and 6 cm thick) such as a beta sialon butterfly valve, large cylinders (330 mm diameter x 500 mm length) and intricate shapes such as turbine rotors, using SFE methods. Sol-gel processing and SFE technology are being systematically developed by the Colloid Research Institute (CRI) in Kita-Kyushu, Yawata Works. Four companies (Nippon Steel, Nippon Steel Chemical Company, NGK Spark Plug Company, and Kurosaki Refractories Company, Ltd.) joined with the Japan Key Technology Center and founded CRI in 1986. New facilities were completed in 1987. Since then, CRI in cooperation with Tohoku University, Tokyo Institute of Technology, Toyohashi University of Technology, Kyushu University and the National Institute for Research in Inorganic Material, and the Institute for Molecular Science has been conducting research on colloid processes. Important contributions are anticipated in the future from this small but competent institute.

Fumihiro Wakai and coworkers at Government Industrial Research Institute, Nagoya (GIRIN) have been investigating superplasticity in oxide ceramics since 1984. Their studies included hot forming of plates, strips, and tubes and joining by solid state diffusion of yttria, zirconia, and alumina. In tensile test strains as large as 200% have been demonstrated and shape forming properties are encouraging. Last year a project consortium consisting of

*The yen to dollar fluctuates almost daily. At the date of these prices 140 yen equals one U.S. dollar.

GIRIN, Suzuki Motor Company, Narumi China Company, Kawasaki Heavy Industry, Riken Company, and Nippon Kagaku Company, was formed to fully explore and exploit superplasticity in zirconia oxide containing ceramic materials. Practical applications are being explored and fundamental studies continued to understand mechanisms of superplasticity. The efforts of this consortium are worth watching.

Higher Strengths, Improved Fracture Toughness and Tailored Properties

Numerous approaches have been taken to achieve higher strength, increased fracture toughness, enhanced delayed fracture, and better environmental resistance of structural ceramics. Progress in the different types of carbides, nitrides, and oxides is discussed below.

Sialons

Sialon materials were discovered in Japan and England (Oyama, and Jack - 1972) independently and nearly simultaneously. By 1983 at least five companies in Japan introduced beta sialon based on licenses from the British firm Lucas Cookson (NGK Spark Plug Company, Kyocera Corporation, Hitachi Metals, Hitachi, Ltd., and Mitsubishi Metals Company). Meanwhile, Japanese companies made concerted efforts to develop their own specialty sialon materials for various applications. Early work on sialon was conducted by Toyota Central Research Laboratory, the National Institute in Research Institute in Inorganic Materials, several government industrial research institutes, and numerous universities. Hitachi Metals Company was one of the early entrants in the race to commercialize sialons. In 1984 they commercialized sialon dies for drawing and extruding copper and aluminum alloys. Later, Hitachi developed a sialon with electrical conductivity using TiN as additive. This material can be finished by means of electrical discharge machining. With the assistance of NIRIM, and support from the Japan Research and Development Corporation, Shinagawa Refractories, Inc., Ltd. developed an alpha-plus-beta type sialon. The company established a pilot plant at Okayama with an initial capacity of about 600 kg per month, and scaled production to about a ton per month in 1986. The company sought to apply this as rollers, extruding and drawing dies and cutting tools. Tokyo Yogyo Company and NKK have improved thermal shock resistance of sialon by producing a sailon composite. Thermal shock behavior was markedly improved by evenly dispersing about 10 weight-percent of hexagonal boron nitride (hBN) with particle diameters of about a micron.

NKK applied this sialon composite for break ring nozzles in a horizontal, continuous casting machine for steel. Field tests demonstrated more than twice the durability compared to a previously tested silicon nitride nozzle configuration. With the sialon-hBN material, continuous casting of up to several hundred meters of steel sheet of plates became possible, compared to 70 meter runs typical of the silicon nitride nozzle brake ring. The new nozzle proved especially useful in continuous casting of stainless steel and high strength steel alloys. NKK used a reaction sintering method based on cold isostatic pressing of Si particles mixed with Al_2O_3 and SiO_2, along with BN additives which is subsequently sintered in nitrogen. The method allows manufacture of complex parts. Initially, NKK produced nozzle rings with external diameters of up to 350 mm but has continued to scale up to larger sizes. NKK, widely known as a steel producer, has been striving to diversify. Considerable effort has been expended in developing hot pressing equipment and engineering ceramics. NKK has been engaged in advanced ceramics research now for more than five years with emphasis on titanium boride(s) and beta sialon.

Last year NKK constructed a pilot plant at Kawasaki and eventually intends to have a sintered silicon nitride production facility (50 tons to 60 tons per year) at its Toyama factory. Alumina, zirconia, and mullite parts will be produced as the market develops. They have invested about 1.5 million dollars in their pilot plant and have constructed two clean rooms for ceramic production; one with 1,000 ppm and another with 10,000 ppm control. The facilities began operation last April with a staff of 13 (seven professionals). NKK expects an initial production of about two tons of sintered ceramic per year. Using technology developed by the Government Research Institute-Kyushu, NKK developed a high strength beta sialon for use at temperatures of 1400°C. The material is being used for air control valves in blast furnaces, skid buttons in reheating furnaces, crucibles, impellers for molten materials, burners and bearings. Currently, they are producing a large butterfly valve (about 25 cm diameter with an attached shaft about 30 cm long and maximum thickness of about 6 cm) of beta sialon which they sell to their subsidiary for about 2.5 million yen, at a profit. The ceramic control valve allows improved temperature control in blast furnace operation, higher temperature capabilities, and longer life at a competitive cost. More than 17 full size valves have been subjected to long-term service testing in full scale heat treatment facilities.

Silicon Carbide

Silicon carbide ceramics received significant attention in Japan for a number of years in spite of the fact that the patent situation was favorable to the U.S. (in particular, for Carborundum-alpha SiC, and GE-beta SiC). Considerable effort was devoted (by Hitachi, Asahi Glass, and others) to finding alternate sintering aids which were superior to boron and carbon for SiC. Major resources were also devoted to investigating numerous processing routes for sintered alpha and beta silicon carbide. By 1987, 13 companies were offering silicon carbide components and three companies were offering high purity powders.

Most recent emphasis has been on ultrafine powders, unique consolidation techniques, and composites. New directions in component processing included hot isostatic pressing (HIP) and SHS. HIP, and HIP combined with self-propagating high temperature synthesis (SHS), has produced high performance silicon carbides. Ultrafine, high purity powders, in the range of 5 nm to 200 nm have been produced via rf or microwave thermal plasmas. Availability of these high purity powders has led to improved properties and the potential for superplasticity effects at high temperature, which are currently being investigated at the GIRI-Nagoya. Abe, and colleagues at GIRI-Nagoya, have been studying fabrication technology, encompassing powder compaction and sintering, microstructure and pore control, effects of firing atmosphere, and influence of sintering aids.

Silicon Nitride

Silicon nitride development in Japan began in the late 1950s. Since then, Japanese researchers have made significant contributions. A major advance was gas pressure sintering of silicon nitride (GPSSN) (developed by Priest & Gazza in the U.S. and Nitomo in Japan) especially the two-step technique (developed in the U.S. by Greskovich at General Electric and Gazza at U.S. Army Materials Technology Laboratory (MTL)). Japan invested heavily to optimize two-step GPSSN for Si_3N_4 and Si_3N_4-SiC composites. Considerable effort has also been spent on HIP of silicon nitrides. Japanese industry has emphasized high purity powders, sintering aids and composite powders, and has achieved considerable improvement in properties. Emphasis has been on improving the high temperature properties and achieving complex shaping capability.

Several teams of investigators have used aluminum nitride (AIN) powders to improve strength and fracture toughness of alpha and beta prime Si_3N_4. In 1986 K. Ishizawa, N. Ayuzawa, A. Shiranita, M. Takai, N. Uchida, and M. Mitomo described such material. In 1987 M. Komatsu, et al fabricated silicon nitride by hot pressing a mixture of Si_3N_4, Y_2O_3, MgO_2 and AIN powders. They reported three point bend strengths of 100 kg/mm^2 at RT, 125 kg/mm^2 at 1300°C and fracture toughness about 6.9. An interesting development in this type of high strength hot-pressed silicon nitride has been reported by Yoshio Ukyo and Shigetaka Wada, Toyota Central Research and Development Laboratory in 1989. The produced a fine-grained sialon composed of alpha and beta Si_3N_4. Their material was formed by hot pressing of Si_3N_4, Y_2O_3, and AIN powders. High strengths of 1.3 GPa at RT and 1.0 GPA at 1400°C, with fracture toughness of about 7.0 $MPa.m^{1/2}$, were achieved. The ceramic microstructure was composed of prismatic grains less than one micron in the long axis. Interestingly, the space between beta prime silicon nitride was filled with finer, rounded grains, ranging in size from 0.1 micron to 0.3 microns. STEM/EDS revealed the prismatic grains and the smaller rounded grains were beta prime and alpha prime silicon nitride, respectively. This particular microstructure resulted in good elevated temperature strength and high fracture toughness as well. High temperature creep, oxidation resistance, and fatigue behavior remain to be explored.

Zirconia

Japan did not play an active role in the early developments of zirconia ceramics. However, Japanese researchers were quick to realize the commercial potential for these materials. Accordingly, they have made major contributions in the optimization of the major classes of zirconia: partially stabilized zirconia (PSZ), tetragonal zirconia polycrystals (TZP), and dispersion toughened zirconia (i.e., zirconia toughened alumina-ZTA, etc.). By the late 1970s Japanese industry was engaged in development of production technology, and by the mid 1980s were major suppliers of high quality yittria stabilized TZP. At lease nine companies were involved with zirconia: Toyo Soda Manufacturing Company, Ltd., and Daichi Kigenso Company, Ltd., were producing Y-TZP powder, and ceramic components were being made by Toray, Kyocera, NGK Spark Plugs, NGK Insulators, Toshiba, Hitachi, Koransha, and Toyo Soda. Kyocera had licensed Feldmuhle's technology for Al_2O_3-ZrO_2(ZTA).

TZP materials can have Y_2O_3 or CeO_2, as well as other additions. Solubility of these oxides in zirconia is much greater than CaO, or MgO, and this diminishes the tetragonal to monoclinic transformation which results in a predominantly tetragonal material. Y_2O_3 or CeO_2 also lowers the temperature at which the tetragonal phase is stable. In this instance the microstructure is fine-grained (about 2 microns) and equiaxed. It has been determined that, for a given temperature, there is a critical grain size needed to prevent spontaneous transformation (t - m) during cooling. Flexural strengths greater than 1.0 GPa, and ranging over 2.5 GPa, have been reported for Y-TZP with toughness varying from 5 to 9 $MPa.m^{1/2}$. HIP was one early way to seek improved Y-TZP. Much of the early work on Ce-TZP was conducted in Japan, and a variety of zirconia toughened materials have been produced through addition of very fine particles of zirconia (generally under 0.5 microns). This involved an active search to improve silicon nitride, mullite, and alumina via dispersion of zirconia. Major industrial thrusts were in powder synthesis, ceramic fabrication, and property evaluation. Meanwhile, university and government labs investigated alternate powder synthesis (CVD, plasma, and hyrothermal among others), transformation toughening mechanisms, high temperature and high pressure fabrication, and property evaluation. Researchers at GIRI-Nagoya, GIRI-Osaka, the Osaka Prefectural Research Institute, as well as at Tokyo Institute of Technology, University of Tokyo, Tohoku University, Osaka University, Ikutoku University, Kyushu University, Kyushu Institute of Technology, Kyoto Institute of Technology and Chubu University contributed to these areas.

Mullite

Japan has taken a lead in producing high strength mullite ceramics having more than twice the strength of ordinary ceramics. The new mullite is being produced using sol-gel and alkoxide powder processing techniques. A number of Japanese firms are producing new mullite powders, and several companies are marketing mullite ceramics. The new mullites are competing for markets formerly dominated by alumina; for instance, laboratory ware, furnace tubes, and substrates. While toughness and thermal conductivity are higher, cost of these mullites are less than alumina. In 1988 Chichibu Cement Company produced mullite furnace tubes as replacement for alumina products. They used a sol-gel technique and sintering to obtain strengths of 350 MPa to 400 MPa. Shinko Electric Company is using mullite for an insulating substrate. Nippon Steel and Kurosaki Yogyo Companies are using a colloid process to make mullite powders and sintering a variety of components for furnaces, machine components, and electronic applications.

Sol-gel Techniques

Sol-gel techniques and colloidal processing are one of the favored methods of shape-forming ceramics. Mitsubishi Mining and Cement Company have been using sol-gel methods to manufacture multilayer capacitors and to produce alumina sheets up to 100 microns thick. Shrinkage is controlled by using microwave drying prior to firing. Researchers at Chichibu Cement Company, in collaboration with Nishi Tokyo University, are using sol-gel techniques to disperse zirconia in mullite. Addition of 15-volume-percent zirconia resulted in bending strengths of 500 MPa and fracture toughness of about 4.3 $MPa.m^{1/2}$ (from RT to 1000^oC). At 1300^oC the values declined to 350 MPa and 2.5 $MPa.m^{1/2}$. The number of companies using sol-gel techniques continues to grow and research activity is at a high level.

Coated and Composite Powders

Numerous organizations have used powder coatings and surface modification techniques to improve properties. Researchers in the Applied Chemistry Department of Nagoya University coated dispersed oxide particles with zirconium alkyoxide by partial hyrolysis. Silica colloidal particles could be easily coated by controlling zeta potential and subsequently sintered to full density. The technique has been applied to zirconia/mullite ceramics as well. Nara Machinery Company has marketed special powder coating equipment and Kyoristu Company, Ltd. is manufacturing a range of composite powders via SHS technology.

Laminated Ceramics

Laminated ceramics have been studied by numerous groups in Japan, including electroceramics manufacturers, universities, government institutes, and materials producers such as Nippon Steel and NKK. These efforts have a variety of purposes such as improved fracture toughness and specifically tailored properties and configurations. Laminated ceramics are being produced via slip casting, injection molding, doctor blade process, sol-gel methods, chemical and plasma vapor deposition, and SHS methods. For instance, Takebe and Morinaga of Kyushu University (1987), have produced and evaluated lamellar alumina ceramics having alternating layers of dense and porous alumina. The lamellar composites were made by slip casting using two types of alumina powders which had different particle size distributions and densification behavior. Researchers at NKK are conducting a systematic exploration of the potential of lamination to produce so-called functionally gradient materials. The work is part of the Science and Technology Agency's project on Research on Fundamental Technology for the "Development of Functionally Gradient Materials Effective in Thermal Stress Relaxation" which was inaugurated in 1987 and was initiated by M. Koizumi (1987). This project aims to develop new functional materials with properties intentionally and continuously controlled along the thickness direction so as to optimize performance under severe operating conditions. The work is conceived to be mainly in support of next generation aircraft and/or spacecraft, but is expected to contribute in other fields. NKK used the doctor blade process to prepare green sheets from slurry made of ceramic-metal powder mixtures of various ratios. Green sheets from several microns to several millimeters thickness were fabricated. During 1988, based on their optimization studies, a prototype pressurization-type green sheet sintering system was fabricated and experimental manufacturing studies continued in development of functionally gradient materials. Laminates of up to five layers were produced with varying metal content. It will be interesting to learn of their achievements in the years to come.

Microstructural and Nanostructural Design

In May 1989 Dr. Koichi Niihara, Physics Department at the National Defense Academy, received the Japan Ceramics Society Annual Award for outstanding research in his studies of toughening and strengthening Al_2O_3, Si_3N_4, and SiC ceramics by microstructural and nanostructural design. Specifically, Niihara incorporated second phases and/or selected structural "defects" into grain boundaries and/or inside grains. In alumina ceramics, he tripled the fracture toughness via additions of superfine particles of SiC and also produced interesting properties via additions of ZrO_2, Y_2O_3, and SiC_w. Strengths of 1.2 GPa were achieved in an alumina composite. He produced silicon nitride ceramics from SI-C-N powder precursors, observing that fine SiC particles were dispersed, not only in grain boundaries, but also in the grains.

Carbon/Carbon Composites

One of the first materials displays the author attended on assignment in Japan was the High Tech Materials Exhibition held at Sunshine City in October, 1985. At that time, a number of companies were exhibiting advanced carbon materials and carbon/carbon composite (C/C) products. Isolite Insulating Products displayed C/C micropore refractories, and Kawasaki Steel, Nippon Steel, and Nippon Carbon exhibited impressive carbon composite materials (large cylinders, tubes, conical frustrum, and plates and sheets). At least a dozen companies are now supplying carbon/carbon materials. More than four companies are producing woven two- and three-dimensional fabrics and preforms with a variety of fibers and mixes of fibers. Nissan Motor Company, Nippon Light Metal Company, and Nippon Carbon Company are marketing carbon/carbon, while Sumitomo Metal Industries and Sumitomo Electric Industries, Ltd., as well as Showa Denko K.K. are making sample shipments. Nippon Steel and Kawasaki Steel Corporation, Kobelco Ishikawajima-Harima Heavy Industries, Kawasaki Heavy Industries, and Mitsubishi Heavy Industries, Ltd. are active in research on these materials. Processing technology encompasses hot pressing of specially impregnated plates, high pressure impregnation of and/or chemical vapor deposition (CVD) and infiltration of preforms. Prices vary from 50,000 yen to over 100,000 yen per kg.

Machining and Ceramics

There are two aspects of machining and advanced materials: the use of advanced materials to enhance machining capability, and improvements in capabilities to machine advanced materials. The two aspects are related. Progress in materials technology has imposed new requirements for precision machining of increasingly difficult-to-machine advanced materials. Advanced materials are the enabling technology to solve the machinability problem. Accordingly, starting in 1980, AIST-MITI initiated a special research project on application of ceramics for machine tools. The effort was headed by the Machining Technology Division in the Production Engineering Department of the Mechanical Engineering Laboratory, Tsukuba Science City.

Machine tool components such as spindles, threaded shafts and ball screws, bearings, chucks and collets, lathe beds, and posts were designed, fabricated, and tested using silicon nitride, zirconia oxides, alumina, and other ceramics and ceramic composite materials. Use of ceramics were shown to substantially improve machining capabilities. A number of ceramics producers and machine tool manufacturers joined the effort, including Kyocera, Nippon Tungsten, Nippon Special Ceramics, Sumitomo Electrical Industries, Mitsubishi Metal Industries, Nippon Insulators, Hitachi Chemicals, and Fujikin Company, Ltd. Ceramic air bearing tables, air slides, screw threads, precision calibration blocks, cutting and milling tools, spinning machine thread guides, ceramic valves, seals, bearings, and springs are all commercially available. However, to date the high cost and difficulty of producing and machining these ceramic machine tool components has been a severe impediment to growth in the machine industry.

As for progress in machining of ceramics, the greatest efforts to make complex shaped ceramic parts have been in near net shape sintering technology. However, there has been a steady effort in high efficiency, ultra precise, complex shape machining. Professor Takeo Nakagawa who is head of the Research Center for Development of Advanced Materials, Institute of Industrial Science, University of Tokyo, has achieved remarkable progress in improving grinding of ceramics. His specialty is the processing of advanced materials and his laboratory is devoted to the fields of machining, grinding, metal forming, molding of metals plastics, and sintering of ceramics. The research laboratories of Professor Nakagawa and Professor Tetsutaro Uematsu, Toyama Prefectural Technical Junior College, jointly developed a grinding wheel and three-dimensional machining center for ceramics. The Nakagawa Laboratory developed a sintered cast iron-diamond powder grindstone. The initial grinding tests were done on three ceramics: alumina, silicon carbide, and silicon nitride. Remarkable cutting depths were achieved and surface roughness of less than 1 micron could also be produced. The initial tests on high density alumina involved cuts as deep as 6 mm and feeds speeds of 10 mm to 3 mm per minute.

Specifications and Standards

Fine ceramics are still in a developing state as an industry, partly due to lack of standard methods of evaluation, lack of consistent nomenclature, numerous technological problems, high cost, and an undeveloped market. Starting in the early 1980s, numerous organizations have assisted MITI in development of standards for ceramics and other advanced materials. Steady progress has been made in developing Japan Industrial Standards (JIS); for instance, a bending strength standard was completed in 1985 (JIS R-1601 at room temperature, JIS R-1604 at high temperature). Since then, standards have been adopted for modulus of elasticity (JIS R-1602), and analysis method for silicon nitride powders (JIS R-1603). In 1986 surveys and research were conducted on tensile strength, in 1987 on fracture toughness and oxidation resistance, and in 1988 on compressive strength. In addition, cooperative work has been underway on friction and wear behavior, corrosion resistance, and creep strength. Japan is active in the Versailles Project on Advanced Materials (VAMAS), one of the international cooperation projects proposed at the Versailles Summit.

An important contribution of Japanese researchers in fracture mechanics was the development of a precracking technique to develop a "natural" crack. In 1986 Nippon Steel researchers, Nose and Fujii, reported a successful technique for precracking ceramics. The method uses a specially devised anvil to load a Vickers indented beam specimen, and is characterized by a pop-in cracking procedure which leads to a reasonably straight, through-the-thickness of the beam crack front. This gives a sharp crack, characteristic of the material being evaluated, and is a prerequisite to accurately measure the inherent fracture toughness. Apparently, the method gives about the same measured fracture toughness values over a wide range of precrack lengths and can be used at elevated temperature as well.

Many of the problems which inhibit utilization of ceramics are also difficulties for other advanced materials. MITI officials in recognition of this fact announced their SUNRISE Project for advanced materials in the spring of 1988. The acronym SUNRISE is based upon: seeds, needs, reliability, intelligence, safety, and economy. The goals of the project are to establish an assessment center, conduct statistical surveys on manufacture and use, create a comprehensive advanced materials data base, achieve standardization of research methods, terminology, products, international exchanges of information, and research cooperation. The categories of materials being considered are new metals, polymers, fine ceramics, and composite materials. The assessment center will interact with existing organizations such as the New Materials Center, the Polymer Center, etc. Twenty-eight characteristics materials features were considered to be common to all advanced materials, and from these 52 items were selected for standards setting scheduled to be completed in the next 10 years.

DEVELOPMENT OF THE INFRASTRUCTURE

Since 1981 there has been a surge of widely different enterprises pursuing development of fine ceramics. These include iron and steel manufacturers, petroleum, nonferrous metals, electronics, and machinery companies. In Japan, over 200 companies participate in fine ceramics; of these, about 90 are producing powders, 47 are producing fibers, and over 125 companies produce parts or components. The overseas partners include: 14 in powders, seven in fibers, and eight in ceramics components. These companies are distributed throughout Japan. Major concentrations of ceramics producers are in Nagoya and Tokyo.

Regarding universities, over 77 academic institutions are engaged in ceramics research. Almost half of this activity is in traditional ceramics, but about 40 universities are contributing towards development of fine ceramics. In the ceramics arena, many researchers from industry join those in the university who are supported by Ministry of Education funds. This provides an important link between academia and industrial interests. There are approximately 250 kozas active in ceramics research. A description of subjects under study by these universities is shown in Figure 1.

SUMMARY AND CONCLUSIONS

Overall Considerations

By 1985 Japan had caught up to the Soviet Union in terms of total expenditures on science and technology. Since then, Japan outstripped the Soviets and West Germany and is entrenched as the second largest economic power and second largest supporter of science and technology in the world. The Japanese had targeted overtaking and surpassing European technology and striving for parity with the U.S. Now, some experts believe that in the 1987-1989 timeframe Japanese technology surpassed U.S. expertise in a number of areas, including broad classes of advanced materials.

Regarding the numbers of research institutions and personnel which serves as a yardstick to measure R&D activities in Japan, in 1988 the Management and Coordination Agency announced that there were 16,263 research institutions. These consist of 88 national and special government funded research institutions, 990 public research institutions, 695 university research institutions, and 14,490 private sector institutions. The total number of researchers is 1,081,000, including 405,000 researchers and 676,000 research assistants. In terms of distribution of percentages of activity, the government and public entities amount to 6.6%, universities are 4.3%, and the private sector is 89.1% of the total.

The following facts are revealing:

- Based on proportionate sizes of population and national economy, almost all aggregate indicators show that now Japan expends money and supports R&D personnel in the same ratio as the U.S.
- The rate of growth of R&D expenditures has exceeded the average increase in gross national product (GNP) for more than 20 years in Japan.
- Private R&D plays a dominant funding role (69% of R&D funding and 67% of R&D performance in 1985).
- In contrast, U.S. industry provided 49% of R&D funds and accounted for 73% of R&D performance in the same year.
- Japanese R&D is 97% to 98% company funded.
- With regard to ratio of company funded R&D to GNP, and nondefense R&D to GNP, Japan's performance has exceeded that of the U.S. for a number of years.
- In textiles, ceramics, and iron and steel, Japanese R&D levels are nearly twice those of the U.S. Especially in ceramics, the science and engineering workforce is larger.
- In all technological areas, Japan's share of U.S. patents granted has increased. The largest gains have been in internal combustion engines (17% in 1975, 44% in 1986) and lasers (14% to 35% in the same years). Other technological areas of extensive activity are semiconductors, computer systems, machine tools, advanced materials, and biotechnology.
- In 1985 Japan had slightly more than 1.5 million employed nonacademic scientists and engineers (S/E) compared to 3.6 million in the U.S. in 1986.

- There has been a continual increase in S/E in Japan. Comparative overall data for engineers in the workforce indicate 187/10,000 in Japan and 183/10,000 in the U.S. There has been a rapid increase in S/E engaged in R&D.
- Japanese S/E are younger; in 1985 50% were under 35 years compared to 28% in the U.S.
- Manufacturing productivity has increased substantially in the past decade in terms of manufacturing output per worker hours; a 68% increase for 1977 to 1986 compared to a 28% increase in the U.S.
- Japan has invested much more R&D in process technology than the U.S.
- Japan's technological balance* of payments have been over a billion dollars per year for a number of years (1.2 billion in 1970 and 1.7 billion in 1985).
- Export of technologically intensive products is increasing.
- Japan is now less dependent on imported technology and now exports significant amounts of technology to newly industrialized nations.

Advanced Materials Considerations

The specific area of advanced materials can be summarized as follows:

In structural ceramics significant advances have been made in producing new powders, preceramic precursor polymers, fiber and whisker reinforcements, and in developing alternate processing techniques for monolithic and composite ceramics. On laboratory scale test specimens, strength levels, creep and oxidation resistance, and fracture toughness have been improved for many varieties of oxides, nitrides and carbides, their composites, and also in c/c materials. In support of U.S. advanced automotive ceramic gas turbine efforts, Kyocera Corporation and NGK Corporation have produced high performance turbine rotors. Other Japanese manufacturers have made significant advances in commercialization of ceramic heat engine components. Considerable progress has been made in use of structural ceramics in the chemicals, steel and nonferrous metals processing industries, for welding and semiconductor heat treatment jigs, cutting tools, and in a host of consumer goods. A large scale national ceramic gas turbine project was initiated in 1988 for a 300 K_w industrial power gas turbine. Future plans include another major Japanese initiative in automotive gas turbines. Old materials have been revisited. Vastly improved versions of mullite and alumina have appeared and interesting combinations of zirconia, alumina, and mullite, with various rare earth additives have yielded impressive high temperature properties. Accomplishments in pitch-based carbon fiber and carbon fiber-reinforced cement are noteworthy. Other significant new directions have been imposed on the community of advanced materials specialists, such as diamond-like materials, high T_c superconductors, functionally gradient materials, and so-called intelligent materials. To an extent, this has diluted the activities on advanced structural ceramic materials, but the predicted market for these new activities is enormous although far from realized.

As for advanced composites, after eight years of effort on MITI sponsored projects, the stated properties goals for plastic and metal matrix composites (FRP and FRM) were met, and in some instances were exceeded. At the peak of the FRP and FRM projects almost

*Import/export balance of trade in high technology products, license, etc.

200 researchers were under MITI support. Now at least 40 new advanced materials tasks are underway in a continuation of the national scale project, Basic Research for Future Industries. Major thrusts are also underway in support of high temperature materials and for various space and advanced aerospace projects. More demanding goals for high temperature materials development have been accepted and are being pursued.

A recent MITI survey of Japanese industry indicated that 70% of the firms responding had in-house R&D efforts and that 65% of these business firms do basic R&D with high tech emphasis. The major goal stated for these activities was to develop revolutionary technology and new products as contrasted to merely improving existing products. Applications development is a premier accomplishment of Japan and process technology R&D is a major activity throughout industry. The magnitude of effort, capital investment, and accomplishments of Japan in materials technology is impressive to Western engineers.

Information exchange in the past two years has been accelerated. There are increasingly viable efforts at international cooperation. Funding levels have been increased and access improved for international exchanges in a wide variety of programs. A number of working groups such as the New Diamond Forum, New Glass Forum, New Carbon Materials, Functionally Gradient Materials, and Self-Propagating High Temperature Combustion Synthesis (SHS) Materials Group, and the Program Management meetings for the National Projects provide a key to the activity in new research areas in Japan.

CONCLUSIONS

New materials activity in Japan is a multifaceted effort: conducting the fundamental research, proceeding into trial applications, investing in the equipment, people, and doggedly pursuing commercialization. The remarkable aspect is the commitment of workers and management to move into the marketplace. Japan is systematically investing and doing the engineering to get the job done. Why has Japan been so successful? Some general reasons are:

- National consensus on important new trends and needed efforts.
- Massive industrial R&D spending.
- Effective system of taxation and subsidized loans to stimulate basic R&D.
- Commitment to nonoil energy, infrastructual, through energy conservation, a strong nuclear energy, and alternate energy programs.
- Recognition of the importance of new materials technology across all sectors.
- Significant cooperative research between companies.
- Willingness to diversify into new areas.
- Effective reconstruction of ailing industries.
- High level of information gathering on worldwide trends in new technology and markets, along with effective sharing and decision making and policy implementation.
- Ability to rapidly exploit the results of R&D.
- High level of interest in technology by the average citizen.

- Highly educated and skilled workforce with excellent work ethic, commitment to teamwork, and with increasing productivity.
- Excellent management with technical expertise and national interests at a premium.

The U.S. and Japan have been vying for dominance in the development of advanced technologies as the mainspring for economic growth. Japan has been successful in institutionalizing and formalizing the importance of science and technology in its economy and very deliberate in setting their priorities and goals. Management of R&D has been particularly effective in playing the game of catchup to the industrialized nations of the world. Now that Japan is on par in the many areas of technology, a moot point is: How good will their decision making, policies, and management be as leaders making major choices for the future? Another major concern is the magnitude of the return on investment for these new materials and emerging technologies. Will the payoff be there?

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1. Fourth International Conference on Structural Safety and Reliability. ICOSSAR 1986, v. 10, no. 3, July-September 1986, p. 116-120.

2. High Tech Materials Exhibition 1986, v. 11, no. 1, January-March 1988, p. 33-60.

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9. A Review of the State of the Art in Rolling Element Bearing Technology in Japan, v. 12, no. 4, October-December 1987, p. 67-70.

10. Survey of Hot Isostatically-Pressed Ceramics, v. 13, no. 2, April-June 1988, p. 33-63.

11. High Temperature Materials Research and Development in Japan, v. 14, no. 3, July-September 1989, p. 43-97.

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3. Review of Ceramics Design and Analysis Procedures, Proceedings of ICAMT Ceramics Workshop: Design, Analysis and Life Prediction for Ceramics, Tokyo, September 1986, p. 43-62.

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9. Interview of R. N. Katz and E. M. Lenoe (in Japanese). Bulletin of Ceramics Society of Japan, November 1986.

10. Smart Materials: Functional Materials for Future Industries. Proceedings (in Japanese) International Symposium on Fine Ceramics - ARITA, Saga Prefecture, November 1988, p. 216-234.

11. Fatigue Behavior of Composite Materials, Poster Session. International Symposium on Composite Materials, Beijing, June 1986.

12. Probabilistic Models of Materials Behavior, 6 pages. International Conference on Computational Engineering Mechanics, ICCEM 1987, Beijing, June 1987.

13. Evaluating Emerging Materials Technology in the Far East. Presented at the 3rd ROK/U.S. Materials Symposium, 31 October - 1 November 1988.

14. Recent Improvements of Hipped Ceramics. Proceedings (in Japanese), 7th HIP Seminar, Kobe, 12-13 November 1987.

15. Design Properties of Ceramics: Current Status and Future Possibilities. Proceedings of Materials Research Society Symposium J, International Meeting on Advanced Materials, Sunshine City, Ikebukuro, Tokyo, 30 May - 3 June 1988.

16. Size Effect in Creep Rupture of Ceramics. Presented at International Conference on Computational Mechanics, Tokyo Institute of Technology, June 1986.

17. Views on Science and Religion: Comparison of Japanese and Western Styles. Proceedings of First Yoko Civilization Conference, Creating the Future of Mankind, Takayama, 31 October - 1 November 1986, p. 70-90.

18. Advanced Composites R&D in the U.S. Army. Presented at Workshop at Korean Agency for Defense Development, July 1987.

WORKSHOPS ON ADVANCED STRUCTURAL CERAMICS ORGANIZED BY ICAMT:

International Committee-Advanced Materials Technology

Workshop No. 1: Design, Analysis and Life Prediction for Ceramics, Tokyo, 18-19 September 1986.

Purpose: Bring together design, analysis, reliability, test, and evaluation specialists to discuss the technology base, including status of techniques, accomplishments, and recent developments.

Workshop No. 2: Characterization of Ceramics and Development of International Standards. Japan Fine Ceramics Center, Nagoya, 9-11 March 1987.

Purpose: The purpose of this workshop was to document the status of characterization techniques currently being applied to advanced ceramics and to explore possible advanced techniques. In addition, the wide ranging international round robin testing activities underway were reviewed.

Workshop No. 3: Status of Centers of Excellence for Advanced Materials. Japan Fine Ceramics Center, Nagoya, September 1987.

Purpose: Document the range of activity at government, industry, and, particularly, university centers of excellence.

Workshop No. 4: Advances in Materials, Processing, and Manufacturing Science. Japan Fine Ceramics Center, Nagoya, March 1988.

Purpose: The sessions provided an overview of major R&D efforts and a forecast of potential limits of materials capabilities in ceramic materials capabilities.